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10/077,658	02/15/2002	Marc Husemann	tesa AG 1525-WCG	2957
27386 7590 06/21/2007 NORRIS, MCLAUGHLIN & MARCUS, P.A. 875 THIRD AVE 18TH FLOOR NEW YORK, NY 10022			EXAMINER COLE, ELIZABETH M	
			ART UNIT 1771	PAPER NUMBER
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EXAMINER

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Commissioner for Patents

Translations of DE 198907752 and DE 4313008 are attached.

Elizabeth M. Cole
Primary Examiner
Art Unit: 1771

Translation of German-Language Patent Document DE. 4,313,008

Inventor: Christian Harder et al.

Priority Document: N/A

Int. Class5: C09J133/06; C09J 7/02; C08 F 6/10;

Application Date: April 21, 1993

Publication Date: November 10, 1994

Original German Title: Selbstklebemasse auf Acrylathomelt-Basis, Verfahren zu deren Herstellung und deren Verwendung

Acrylate Hot-Melt-Based Self-Adhesive Composition

The invention relates to an acrylate hot-melt-based self-adhesive composition having a K-value of at least 60.

On the one hand, acrylate hot-melt-based self-adhesive compositions are known. But due to their low K-value, they are not suitable or inadequate for many applications. For example, commercially available compositions of this kind have low viscous systems, such as HRJ 4326 (Schenectady Chemicals) which has a K-value of 31 (testing method by Beiersdorf) and a viscosity of 3.8 Pa·s at 177°C (350°F), or ACRONAL DS 3429 X (BASF) having a K-value of 40 and a viscosity of 13 Pa·s at 120°C. These and similar compositions have not been suitable for coating woven and non-woven fabrics or as substrates for self-adhesive tapes or band-aids, since they penetrate these types of substrates. Moreover, their adhesive capacity is too low for technical applications.

The limitation of these types of known compositions having low viscous systems is especially due to the fact that the required reconcentration step makes high demands on flow behavior and the durability with respect to heat and shear. This is why the adhesives made in the solution are usually reconcentrated in the tank. This method requires:

- low viscosity of the system even in the highly concentrated state in order to effectively maintain the degassing and mixing processes;

- low viscosity of the compositions in order to be able to remove them from the tank in an economical manner.

For the reasons given above, it has previously not been feasible to manufacture acrylate hot-melt adhesive with higher K-values, but compositions having a lower viscosity are afflicted with weak adhesive properties with respect to shear strength.

It is the object of the present invention to eliminate these drawbacks, especially with respect to self-adhesive composition having an acrylate hot-melt basis and a K-value of at least 60.

Such compositions are not commercially available, especially because no technologically feasible and meaningful method exists for manufacturing the same. This is the case, despite a significant interest in such compositions and extensive developmental work in order to increase the K-values of the compositions, which have until now not been offered commercially

The problem under consideration is solved by the compositions and their manufacturing process as defined in the main claims.

The use of a degassing extruder to solve the problem under consideration was not within the field of vision of the person of skill in the art for a plurality of reasons: It was previously supposed that high K-values of the acrylate hotmelt, i.e., at least 60, together with degassing would result in the decomposition of macromolecules due to shear forces and heat treatment, and gelling would occur due to reactive Co components in addition to the basic concern about too high a content of solvents, and as it turns out, the prejudgment against using a degassing extruder at all for this type of high molecular and adhesive products. Although it has been known from European Patent EP-PS 411,510 to remove volatile components from a polymer solvent in the extruder, which is done especially to remove residual monomers. Although the process described in this European Patent, involves, as a usual in prior art, different thermoplastic substances such a polycarbonate, polyphenylene ether or polystyrol, they are not comparable to the inventive acrylate hot melts having a high K-value as employed in accordance with the present invention. Because in the judgment of

the person of skill in the art, adhesive compositions cannot be transported in a degassing extruder.

In accordance with the invention it is preferable to employ the measures as defined in the dependent claims. Solvents employed in this case are: benzene, acetone, acetate, toluol, C1-C5- alcohols, C5-C9 alkanes and mixtures thereof. Additional measures may be taken for the reconcentration process, for example, entrainers, such as water, may be used to accelerate the degassing process. Inert gas, such as nitrogen or argon, may also be used to break the vacuum. It is especially advantageous if the extruder employed in this case is provided with a plurality of heatable zones which are independent of one another. The transport performance is also adjusted by altering the rpm and the helical configuration to comply with the acrylate hotmelt being processed.

This resulted in advantages which could not have been anticipated by the person of skill in the art:

- compounds having a high K-value can be degassed while allowing non-abrasive removal because the temperature and shear strength can be adjusted as required. Limitations with respect to formulation possibilities are eliminated with respect to the low-viscous systems.

- the flow of the composition in the 100% state is not absolutely necessary due to the mechanical transport;

- in contrast to the batch method, continuous production is possible;

- compositions having a high K-value also fulfill the requirements for a technical adhesive;

- shear strengths of these systems are higher, while the required refining process is simplified.

The invention is further elucidated by way of the exemplary embodiments without unnecessarily limiting the same.

Example 1

The following monomer mixtures (quantities given in wt %) were copolymerized in a solution. The polymerization composition comprised 60 wt.-% of the monomer mixtures and 40 wt % solvent.

Oxygen was first removed by rinsing with nitrogen in the usual reaction vessels made of glass or steel (including backflow coolers, stirrers, temperature measuring unit, and gas inlet pipe) and then heated to boiling point. By adding the usual initiators such as peroxides or azo initiators required for the radical polymerization, the polymerization was initiated.

During the polymerization time of approximately 20 hours, further solvents were used, if indicated, to further dilute the composition several times as a function of the viscosity in such a way that the finished polymer solution had a solid substance content of approximately 35 to 55 wt %.

A 40% solution of the acrylic acid(ester) copolymerizate having a K-value of 68 (76% ethylhexyl acrylate, 21% n-butylacrylate, and 3% acrylic acid) in the mixture comprising benzene (60/95) and acetone at a ratio of 3:1 were separated by means of a single-screw extruder (d=90, l=38d) into polymer and solvents. The extruder comprised a total of three independently heatable zones of which each was provided with a vacuum connection and an inlet for nitrogen. The vacuum devices for the zones were also adjustable independent of one another, both by way of the vacuum pumps connected downstream, and the control unit for the nitrogen supply. The polymer solution was transported through a pump to the extruder between the first and second zone in such a way that the gas phase was removed via the polymer solution in one backflow and two forward degassing steps. The inflow of the polymer solution was 18 kg/h, and the rotational speed of the screw 100 U/min. The temperature of the backflow degassing was 60°C at a pressure of 270 mbar, with the temperature of the second and third step being adjusted to 40 or 50°C under pressures ranging from 310 and 40 mbar, respectively. The gel-free polymers were removed from the top of the extruder with a composition flow of 7.2 kg/h. The proportion of volatile substances in the polymers was 0.8%, and the K-value was 68.

The acrylate hotmelt obtained in this manner can be applied in a known manner to coat woven substrates without penetrating the same, and the resulting products have excellent adhesive properties.

Example 2

A 45% solution of this acrylate acid(ester)copolymer having a k-value of 65 (33% ethylhexyl acrylate, 64% butylacrylate, and 3% acrylic acid) in acetone was reconcentrated using the method described in Example 1. The component of the volatiles substances was 0.85, the K-value was 65.

The composition can be mixed with solid glass micro spheres in accordance with European Patent EP 431,336 and processed by a known method into products having excellent adhesive properties.

CLAIMS

1. Self-adhesive composition on an acrylate hotmelt base, characterized by a K-value of at least 60.
2. Self-adhesive composition as defined in claim 1, characterized by a K-value ranging from 65 to 80.
3. Self-adhesive composition as defined in claim 1, characterized in that copolymers consisting of (meth)acrylic acid and esters thereof having 1-25 C atoms, maleic, fumaric and/or itaconic acid and/or esters thereof, substituted (meth)acrylamides, maleic acid anhydride, and vinyl esters, more particularly vinyl acetate, vinyl alcohols and/or vinyl ethers are used.
4. Self-adhesive composition as defined in Claim 1, characterized in that the content of the residual solvent is less than 1 wt %.
5. Method for manufacturing a self-adhesive composition on an acrylate hot-melt base having a K-value of at least 60 as defined in one of Claims 1 to 4, characterized in that a solution of said composition is reconcentrated in an extruder by a degassing process to obtain a composition that can be processed into a hotmelt.
6. Method as defined in Claim 5, characterized in that the solution of the composition contains 5 - 80 wt % more particularly 30 - 70 wt % solvents.

7. Method as defined in Claims 5 or 6, characterized in that commercially available solvents are used, more particularly, hydrocarbons, ketones, alcohol, and/or esters having a low boiling point.

8. Method as defined in one of Claims 5 to 7, characterized in that single-screw, dual-screw, or multiple-screw extruders having one, more particularly two, or a plurality of degassing units are used.

9. Utilization of the self-adhesive composition as defined in one of Claim 1 to 4 on articles that are provided with self-adhesive compositions, such as band-aids or self-adhesive tapes.

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Translations Branch - Martha Witebsky
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